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Also reported are the results from field and laboratory studies on the reliability of using individual carbon dioxide and oxygen analyzers of the Fyrite* type to determine stack gas molecular weight. The laboratory study, which was done using three cylinders containing mixtures of carbon dioxide, oxygen, carbon monoxide, and nitrogen of known concentration, determined that these analyzers give carbon dioxide and oxygen analyses of sufficient reliability to yield accurate molecular weights. The results of the field studies, which were done on actual flue gas samples, also support this conclusion.

*Reference to trade names on commercial products does not constitute endorsement by the Environmental Protection Agency.

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Field Reliability of the Orsat Analyzer

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U.S. Environmental Protection Agency

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Under the provisions of Section 111 of the Clean Air Act as amended in December 1970 the Environmental Protection Agency (EPA) is authorized to establish standards for emissions of air pollutants from stationary sources (performance standards). The first set of standards, which contained nine *Test Methods* for determining emissions from new stationary sources, was promulgated in December 1971. One of these nine test methods, *Method 3*[1]*—Gas Analysis for Carbon Dioxide, Excess Air and Dry Molecular Weight*, required use of the Orsat analyzer.

Traditionally, Orsat data have been used to obtain the following information: 1) the dry molecular weight of the stack gas, and 2) the multiplication factor required to convert particulate loadings from combustion sources to such reference conditions as 12% CO_2 and 50% excess air. For example, EPA requires that the Orsat analytical procedure described in Method 3 be used to correct the measured particulate loading

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from municipal incinerators to the reference condition of 12% CO_2.

At the time Method 3 was promulgated, the within-laboratory and between-laboratory precision of the Orsat analytical procedure under field conditions was unknown. Thus, when Method 3 was promulgated, EPA attempted to compensate for the uncertainty about the precision of the Orsat procedure by including in the procedure an operator performance criterion. This criterion must be met before Orsat data can be used to convert particulate loadings at municipal incinerators to the reference condition of 12% CO_2. The criterion requires that the integrated (bulk) flue gas sample be analyzed until three consecutive analyses vary from each other by no more than 0.2% by volume for each component being analyzed.

The major purpose of our collaborative studies was to determine the within-laboratory and between-laboratory precision associated with the Orsat analytical procedure when personnel experienced with the Orsat analyzer use this procedure in the field. From this data, we could then evaluate: 1) the usefulness of the operator performance criterion in assuring that good data is obtained and 2) the validity of using Orsat data to convert particulate loadings to 12% CO_2 and 50% excess air.

An additional purpose of the study was to determine whether CO_2 and O_2 analyzers of the Fyrite type were suitable substitutes for the Orsat analyzer in determining stack gas molecular weight. Major disadvantages in using the Orsat analyzer in the field are: its fragile construction, the large number of connections, the elaborate leak check procedure necessary before using the equipment, and the number of times the sample must be passed between the buret and the pipet in order to obtain complete absorption. The Fyrite type analyzer is more suitable for field use, because it is inexpensive, light, rugged and of simple construction, capable of being held in one hand, and allows analyses to be done rapidly. For example, three CO_2 and three O_2 analyses can easily be done in less time than it takes to do one complete Orsat analysis. (At this time, a Fyrite type CO analyzer is not available.)

Experimental

Test Sites

The four, field based collaborative tests of the Orsat procedure were conducted at three individual sites—a coal-fired power plant and two different municipal incinerators. Tests 1 and 4 were both conducted at the same site at different times. Test 5, which was a laboratory based collaborative test, was done at an EPA source simulator located in Research Triangle Park, NC.

Description of Equipment Checks

Prior to each collaborative test, the appropriate, freshly prepared absorbing solution was added to each pipet. Then the stopcocks on each Orsat were leak checked at the site. The buret stopcock was leak checked by: 1) displacing the meniscus of the buret liquid until it appeared in the graduated portion of the buret; 2) closing the exhaust valve; 3) placing the leveling bottle on top of the Orsat; and 4) observing the meniscus for movement over a 5 min period. If a volume change of less than 0.4 ml occurred during this 5 min interval, the apparatus was considered to have passed the leak check. Each pipet stopcock was checked for leaks by: 1) bringing the pipet solution up to the reference mark on the capillary; 2) closing the pipet/manifold stopcock; and, 3) observing the liquid level for movement over a 5 min interval. If the liquid level fell below the reference mark during this time, the pipet failed the leak check. If a stopcock failed the leak check, it was disassembled and cleaned, reassembled, and then checked again. If the rubber connecting tubing was the source of the leak, it was replaced. (Because the Orsats were not leak checked at the conclusion of the collaborative tests, the possibility that leaks developed during the test cannot be evaluated.)

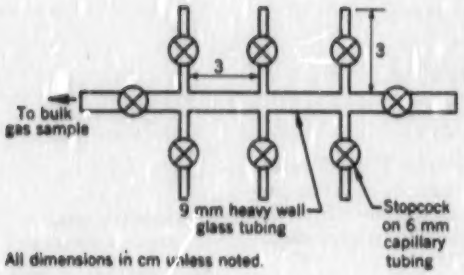
Design of the Collaborative Tests

Collaborative Tests 1 and 2 were designed to evaluate the precision of the Orsat analyzer under field conditions and to determine the capabilities of the Orsat operators to meet the EPA performance criterion previously discussed. In Test 1, 4 participants each analyzed 7 sample replicates from the same bulk (integrated) flue gas sample. In Test 2, 6 collaborators each analyzed 4 replicates from the same integrated flue gas sample.

Tests 3 and 4 were designed to study the differences observed between operators in the first two tests. Test 3 was designed to see if improving the "readability" of the buret would improve operator precision. Special 100 ml burets were constructed for this test. These burets differed from the 100 ml burets used in Tests 1 and 2 in four ways: 1) they were graduated from 0 to 25 ml instead of from 0 to 50 ml; 2) the burets in this test were graduated in 0.1 ml intervals instead of in 0.2 ml intervals as in Tests 1 and 2; 3) the buret length equivalent to a 1.0 ml volume change—hereafter referred to as the buret Δ ratio—was 11.0 mm for the Test 3 burets compared to the 4.0 mm/1.0 ml volume change of the burets used in Tests 1 and 2; and 4) as an aid in avoiding parallax, the scribe line at each full milliliter mark on the burets completely encircled the buret and not just a third of the buret circumference. Four replicate samples were analyzed from the same integrated flue gas sample by each of 5 collaborators using these modified burets.

The fourth test attempted to determine if changing only the buret graduation from 0.2 to 0.1 ml would improve the agreement between operators. In Test 4, the results obtained with standard burets graduated in 0.2 ml intervals (Δ = 4.0 mm/ml) were compared directly with the results obtained with commercial burets graduated in 0.1 ml intervals (Δ = 5.5 mm/ml). These were designated as Tests 4a and 4b, respectively. The assumption was made that the small differences in the Δ ratios of these burets would be relatively insignificant, and that any effects observed would be primarily the result of differences in the buret graduations. Five replicate samples were analyzed from the same integrated sample by each of 4 participants using each type of buret.

The 4 field collaborative tests were designed so that within the same test, all the participants would analyze the same number of replicates. Under this restriction, few of the participants were able to achieve the operator performance criterion even once. Thus, no conclusion could be made about the precision that would have been obtained if only field data that met the performance criterion was used in the statistical analysis. So, a fifth collaborative test, which used standard Orsat apparatus, was done to determine whether meeting the operator performance criterion would decrease the observed differences between Orsat operators. In this fifth test, the 7 participants analyzed 3 integrated flue gas samples until they were able to meet the EPA operator performance criterion on each integrated sample. This usually required not more than 4 replicates, and, only in two cases were as many as 6 or 7 replicate analyses required. Only data that met the performance criterion were used in the statistical analysis.



All dimensions in cm unless noted.

Figure 1. Sampling manifold.

Sampling and Analysis

In all 5 collaborative tests, the integrated flue gas samples were connected to a 7 port sampling manifold (Figure 1). Each participant then connected his apparatus to this gas sample using one of the ports on the manifold. To ensure that the integrated sample was not contaminated by leakage of air into the manifold through one of the Orsats, the stopcock on each port of the manifold was opened only while a sample was being withdrawn from the bulk sample.

Comparative Study of Fyrite and Orsat Analyzers

The field-based Fyrite studies were done on the integrated gas samples used in the four Orsat field-based collaborative tests. The laboratory based study was done on three gas cylinders containing known concentrations of CO_2, O_2, CO, and N_2. Each cylinder was analyzed three times for CO_2 and three times for O_2 using Fyrite type CO_2 and O_2 analyzers. Prior to conducting this experiment, the cylinder manufacturer's gas chromatographic analyses of the three gas mixtures was checked using an Orsat analyzer equipped with one of the burets used in test 3. The Orsat results on each gas mixture agreed within ±0.2% of those obtained by the manufacturer.

Results

The original field data are too voluminous to present here, but they are available from the authors upon request. The mean values for percentage CO_2 and percentage O_2 obtained by each participant in the 4 field based collaborative tests are presented in Table I. Mean values for percentage CO are given only for Tests 1 and 4. In Tests 2 and 3, no attempt was made to analyze for percentage CO, because a preliminary Orsat analysis of the integrated sample had detected no CO. The mean for each participant was calculated using all the values obtained by that participant.

Table II presents the mean values for percentage CO_2 and percentage O_2 obtained by each participant in the laboratory-based collaborative test. In this test, three integrated gas samples, each containing a different CO_2/O_2 ratio, were analyzed by each participant. The mean values presented were calculated using only those values that met the EPA Method 3 operator performance criterion for a valid Orsat analysis.[1]

Estimates of the variation among laboratories, calculated as between-laboratory standard deviations, are presented in Table III for the five collaborative tests. In addition, a within-laboratory standard deviation is presented for each of the 4 field-based studies. A within-laboratory precision was not calculated for Test 5, because the restriction of using only those values which met the EPA Method 3 operator performance criterion would render such a term meaningless when applied to the Orsat analysis itself. No precision estimates were calculated for CO, because of the limited amount of data and the fact that some of these values were zero. All estimates were calculated on the assumption that each collaborator represented an independent laboratory. This is a reasonable assumption, because each collaborator conducted his analyses independently of the other collaborators. [The results from Collaborator D (Test 1) and Collaborator F (Test 2) were not used in calculating these estimates, because Cochran's test for homogeneity of variances[2] determined that the variances obtained in their analyses by these collaborators were statistically different from those of the other participants in the respective collaborative tests.]

The molecular weights (M.W.) and the conversion factors (C.F.) for 50% excess air and for 12% CO_2 in Table IV were calculated from the field based collaborative tests. The conversion factors for 50% EA and 12% CO_2 were calculated using

Table I. Participant means[a] in the field based collaborative tests.

Test No.	Test Conditions	Participant	Means % CO_2	Means % O_2	Means % CO
1	0.2 ml buret	A	2.94	16.80	0.46
	graduations;	B	3.14	16.69	0.68
	Δ = 4.0 mm/ml;	C	3.04	17.02	0.06
	seven replicates	D	2.99	16.90	0.07
2	0.2 ml buret	E	12.75	6.08	—
	graduations;	F	10.10	5.58	—
	Δ = 4.0 mm/ml;	G	12.55	5.45	—
	four replicates	H	12.40	5.15	—
		I	12.90	5.20	—
		J	13.05	5.60	—
3	0.1 ml buret	F	5.02	15.08	—
	graduations;	G	4.88	15.10	—
	Δ = 11.0 mm/ml;	H	5.20	15.05	—
	four replicates	I	4.63	14.68	—
		K	4.80	15.10	—
4a	0.2 ml buret	L	2.08	16.45	0.56
	graduations;	C	2.32	17.42	0.10
	Δ = 4.0 mm/ml;	M	2.56	18.04	0.00
	five replicates	B	2.42	18.16	0.10
4b	0.1 ml buret	L	2.18	16.92	0.24
	graduations;	C	2.46	17.48	0.06
	Δ = 5.5 mm/ml;	M	2.54	17.80	0.02
	five replicates	B	2.64	18.08	0.00

[a] Mean of all values obtained by that participant.

equations 1 and 2, respectively.[3] (In Eq. 1, the CO_2 and O_2 values are expressed in units of mole fraction.)

$$\text{C.F.(50\% EA)} = \frac{(CO_2) + 1.75}{2.88 - 13.9(O_2)} \quad (1)$$

$$\text{C.F.(12\% } CO_2\text{)} = \frac{12\%}{\text{Mean \% } CO_2} \quad (2)$$

The molecular weights presented in Table IV were determined by calculating the molecular weight for each analysis by each participant, and then calculating the mean from the individual molecular weights. The molecular weights themselves were calculated using equation 3.[1]

$$\text{M.W.} = 0.44(\%\ CO_2) + 0.32(\%\ O_2) + 0.28(100 - \%\ CO_2 - \%\ O_2) \quad (3)$$

Table V shows the results obtained on three gas mixtures of known concentration using the Fyrite type CO_2 and O_2 analyzers. The Fyrite field results yielded molecular weights that agreed within ±0.2 g/g mole of the mean molecular weight from the Orsat data and, therefore, these results are not presented in the table.

Discussion of Results

From Tables I and II, it is evident that measurable differences in participant means were common in all five collaborative tests. An inspection of the participant means in Tests 4a and 4b (Table I) shows that the relative ordering of the data for individual analysts for percentage CO_2, O_2, and CO remained approximately the same when the buret graduation was changed. Also in both tests, participant L reported significant quantities of CO, but the lowest values for percentage O_2 and percentage CO_2. Looking back to Test 1, it is also seen that participants A and B reported significant CO concentrations in conjunction with the lowest concentrations of O_2 for that test. Because the CO absorbing solution can also absorb O_2, it seems quite possible that these phenomena resulted because of the incomplete absorption of O_2 by its absorbing reagent. Incomplete absorption of CO_2 by its absorbing reagent could also be occurring, but probable instances of this are not so readily identifiable. Overall, the results demonstrate that the Orsat procedure is extremely sensitive not only to operator training, but also to the care and patience the operator takes in the analysis.

Table II. Participant means[a] in the laboratory based collaborative test.

Gas Component	Participant	Means		
		Level 1	Level 2	Level 3
% CO_2	N	2.20	4.40	7.80
	O	2.17	5.33	8.13
	P	2.03	4.83	7.97
	Q	2.60	5.60	8.80
	R	—	5.00	7.85
	S	2.10	5.20	8.20
	T	2.23	5.33	8.23
% O_2	N	17.70	14.30	8.80
	O	17.60	13.00	8.60
	P	18.00	13.27	8.23
	Q	18.60	13.70	8.33
	R	—	12.70	7.67
	S	17.67	13.00	8.80
	T	17.60	12.97	8.53

[a] Mean of the three analyses that met the operator performance criterion for a valid Orsat analysis.

Table III. Estimates of the precision of the Orsat procedure.

	Test	Buret Δ Ratio	Gas Component	
			% CO_2	% O_2
Within-laboratory standard deviation	1	4.0	0.39	0.80
	2	4.0	0.55	0.49
	3	11.0	0.18	0.14
	4a	4.0	0.29	0.45
	4b	5.5	0.31	0.35
Between-laboratory standard deviation	1	4.0	0.39	0.80
	2	4.0	0.55	0.57
	3	11.0	0.27	0.22
	4a	4.0	0.32	0.87
	4b	5.5	0.35	0.59
	5			
	(Level 1)	4.0	0.20	0.38
	(Level 2)	4.0	0.39	0.55
	(Level 3)	4.0	0.34	0.41

From Table III, it is evident that the best overall operator precision in the field based collaborative tests was obtained in Test 3—the test in which the buret Δ ratio was two to three times larger than in the three other tests. (For a discussion of the Δ ratio see the description of Test 3 under "Design of the Collaborative Tests.") In comparing Tests 4a and 4b, it can be seen that the precision was not appreciably affected by the buret graduation itself. These two observations indicate that improving the "readability" of the buret by increasing the Δ ratio can improve the precision of the Orsat analytical procedure.

A comparison of the precision estimates in Table III shows that the between-laboratory standard deviations were somewhat smaller for the laboratory based study than for the field based studies. However, the laboratory based values are still sufficiently large to indicate that meeting the operator performance criterion will not by itself assure that valid Orsat data will be collected. The operator performance criterion was usually met in the laboratory on the first 3 or 4 replicates, but it was rarely met by anyone in the field when allowed from 4 up to 7 replicates. This indicates the performance criterion is more difficult to achieve under field conditions.

Each participant's conversion factor for correcting particulate emissions to 12% CO_2 is shown in Table IV for the field based studies. In examining the range of reported values in those tests in which the conventional Orsat analyzer with the standard buret was used (Tests 1, 2, 4a), it becomes apparent that differences on the order of 5 to 20% would have occurred if each operator within a test had used his mean percent CO_2 result to convert an identical particulate catch to 12% CO_2. And these calculations were made without the extraneous value of participant F in Test 2! In looking at the results of Test 3, which used burets with a larger Δ ratio, one finds a range of about 12% in the conversion factors. However, without a direct comparison, which could only be obtained by using both the standard and the modified burets on the same sample under the same conditions, it is impossible to say precisely just what effect improving the "readability" of the buret alone would have on the spread in the reported values for this conversion factor. Since it was the primary intent of this report to address the performance of the Orsat analyzer under field conditions, such calculations were not carried out for Test 5.

The results for the 50% excess air conversion factor are analogous to those obtained for the 12% CO_2 conversion factor. That is, if each participant within a test had used his percent

oxygen results to correct an identical particulate loading determination to 50% excess air, errors on the order of 4 to 35% would have been introduced into the particulate determination.

When we examine the molecular weights determined on the same sample by each participant (Table IV), the wide differences in mean values between participants (Table I) are obviously not reflected in the average molecular weight determined by each participant. This suggests that if the Orsat results are used for determining only flue gas molecular weight, a performance criterion based on the molecular weight determination itself will be more realistic than one based on the analytical precision for each gas component.

Table V presents the Fyrite results obtained from the analyses of three gas mixtures that are representative of flue gas samples. The excellent agreement between the Fyrite results and the "true" concentration (manufacturer's analysis) support the conclusion that, for determining molecular weights, combustion gas analyzers of the Fyrite type are suitable substitutes for the Orsat analyzer. This is true even in the presence of CO, which apparently does not interfere in the percentage CO_2 and percentage O_2 analyses. (Two of the cylinders contained approximately 1% CO.)

Conclusions

The results from 5 collaborative tests of the Orsat Method indicate that the use of Orsat data to determine the molecular weight of flue gases is a valid procedure, but the use of such data routinely to convert particulate catches to such reference conditions as 12% CO_2 and 50% excess air may introduce sizeable errors in the corrected particulate loading. Further, the EPA operator performance criterion[1] does not ensure that accurate results will be obtained.

However, since the use of Orsat data for calculating particulate conversion factors will likely continue, it seems prudent to develop procedures to check the reliability of Orsat data. One procedure, that could be instituted without affecting either the cost or time of a source test, would be to require that if the Orsat data are to be used for calculating a particulate conversion factor, then the integrated flue gas sample must be independently analyzed by at least two analysts and their results for each gas component must agree within a certain volume percent—say 0.3%—before they can be used to calculate the conversion factor.

Work should also be done to improve the "readability" of the Orsat buret. For example, the column of the bulb above the graduated portion of the buret could be increased by 25 ml so that the graduated portion spanned only 0 to 25 ml (in 0.1 ml divisions) and the space saved could be used to increase the buret Δ ratio. This change is feasible, because most gas streams contain about 80% nitrogen and nitrogen is determined in the Orsat procedure only by difference. Also, the opportunity for parallax to occur could be reduced by completely circumscribing each full milliliter mark around the buret.

It is also evident from these collaborative tests that if an operator performance criterion is to be applied to molecular weight determinations obtained from Orsat data, then it would be more realistic to base the criterion on the molecular weight and not on the analysis for each gas component.

Table IV. Mean dry molecular weights (M.W.) and conversion factors (C.F.) for 50% EA and 12% CO_2.

Test	Participant	M.W.	Conversion Factors	
			50% EA	12% CO_2
1	A	29.1	3.3	4.1
	B	29.4	3.2	3.8
	C	29.2	3.5	3.9
	D	29.2	3.5	4.0
2	E	30.3	0.92	0.94
	F	29.8	0.88	1.2
	G	30.2	0.88	0.96
	H	30.2	0.87	0.97
	I	30.3	0.87	0.93
	J	30.4	0.90	0.92
3	F	29.4	2.3	2.4
	G	29.4	2.3	2.5
	H	29.4	2.3	2.3
	I	29.4	2.1	2.6
	K	29.3	2.3	2.5
4a	L	29.0	3.0	5.8
	C	29.1	3.9	5.2
	M	29.1	4.8	4.7
	B	29.1	5.0	5.0
4b	L	29.0	3.4	5.5
	C	29.1	3.9	4.9
	M	29.1	4.4	4.7
	B	29.1	4.8	4.5

Table V. Fyrite results on gas mixtures of known concentration (in nitrogen).

Cylinder	Manufacturer's Results				Fyrite Results[a]		
	% CO_2	% O_2	% CO	M.W.	% CO_2	% O_2	M.W.
1	1.9	17.9	1.0	29.0	2.0	17.5	29.0
					1.8	18.1	29.0
					1.8	18.0	29.0
2	5.0	14.8	0.0	29.4	5.0	15.3	29.4
					5.0	15.2	29.4
					5.2	15.2	29.4
3	12.9	7.2	0.9	30.3	12.8	7.3	30.3
					12.7	7.0	30.3
					12.7	7.2	30.3

[a] No Fyrite analysis for CO performed; apparatus for measuring CO not yet available.

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